RESEARCH ARTICLE

Adsorptive Capacity of Coconut Fibre Carbon Activated by Potassium Hydroxide for Wastewater Treatment

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ABSTRACT: Optimization of the yield and adsorptive effectiveness of activated carbon prepared with coconut fibre and potassium hydroxide (KOH) for the treatment of wastewater is presented. Different values of the operating parameters (activation temperature, X_1 and impregnation ratio, X_2) in the range of 350 to 550^{o}C and 1 to 3g/g for an activation time of 5 min were used to investigate the activated carbon yield. The study used wastewater laden with iodine to ascertain the prepared activated carbon's capacity to absorb its iodine content. The experimental results obtained showed the values of activated carbon yield (Y_1) to be in the range of 3.99 to 17.45%; whereas the iodine adsorption capacity (Y_2) ranged between 44.140 and 192.070 mg/g, respectively. It was observed that when the activation temperature was kept constant and the impregnation ratio was increased, the activated carbon yield increased gradually and then dropped. The same trend was observed when the impregnation ratio was kept constant, while the activation temperature was varied. An optimization study was carried out to determine the combined effect of X_1 and X_2 on the values of Y_1 and Y_2 which were to be maximized to determine the optimum conditions for activated carbon yield and its adsorptive capacity for wastewater treatment. The results of the best fit achieved for all 10 possible runs showed optimum values of activation temperature, impregnation ratio and adsorption capacity as 354.592^{o}C, 1.420g/g, and 4.830 mg/g, respectively. A suggestion for further studies is given.

KEYWORDS: Wastewater, Adsorbent, Treatment, Coconut Fibre, Carbon, Optimization.

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1. INTRODUCTION

Activated carbons (CA) are those carbonaceous substances with a large surface area and a high degree of surface reactivity, which indicates a high adsorbing capacity, which is a result of the numerous pores on their surface [1]. Such materials are mostly made from lignocellulose-based materials and other carbon-rich substances such as coal [2,3]. A few examples of agricultural wastes that are lignocelluloses-based materials that are now in use as activated carbons production precursors are palm kernel shells, corn cobs and coconut shells [4-6]. In the metal extraction industry, gas and liquid phase industries now use activated carbons as adsorbents. Furthermore, the most important industries in terms of application and consumption of activated carbon are small and movable waste treatment plants, with a strong affinity for heavy metals, which when not removed, can be very harmful to human life [7,8].

Conventionally, activated carbon materials are prepared by using the method of either chemical or physical activation or both. And in any of the methods, a process involving two stages is usually considered in their production which includes an initial carbon content enrichment for the production of a carbonaceous mass that has many tiny pores

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[4]. This is followed by chemical or physical activation. For the physical activation option, the char that results from the process is activated using selective oxidation in the presence of a good gasifying agent such as steam or CO_2 at temperatures in the range of 600 to 900^{o}C [9]. It is on record that CA is among the most commonly utilized adsorbents for wastewater treatment on a global scale. As a result of its porous nature and parent derivative origin, it eliminates natural organic components, flavour and odour constituents, as well as manmade organic substances from water and air, thus, making it an essential component of the filtration process. Between the early 60s and 70s, the developed nations of the world started to utilize the activated carbon technique to increase the elimination of organic pollutants from waste water for different purposes. In affluent nations such as the United States, Japan, the Netherlands, and Switzerland, among others, the activated carbon technique has become the most prevalent kind of modern treatment of waste water [10]. The frequent global application of CA in the treatment of waste water, drinking water, catalyst aid, as well as gas decontamination relies on its high absorption aptitude [11]. The adsorption process links molecules in the gaseous or liquid phase to the CA's surface. This renders it an efficient adsorbent agent given its high porosity and larger surface area on which impurities can be adsorbed.

Coconut fibre, as an agricultural waste has been selected in this research because it is readily available with little value in rural and urban areas. Thousands of tons of coconut fibre wastes are generated yearly in Nigeria by rural farmers and there are projections for an increase in the subsequent years due to the rapid emergence of enhanced growth species of coconut now available in various localities [12] and the need to meet the required standard for wastewater treatment as prescribed by the United Nation's development goals - which is to have global and adequate access to inexpensive, safe drinking water for everyone by 2030 to avert the perilous human and environmental effects of untreated wastewater.

It is pertinent to recall that KOH is one of the inorganic compounds that, despite not being carcinogenic, is harmful to living creatures such as aquatic life in very low concentrations. Hence lethal dosage should be monitored when the process comes to real-time applications by neutralizing with dilute acid (like acetic acid). It is required in quite significant quantities by plants that are growing. Potassium deficiency results in a range of indicators, including stunted growth, diminished flowering, decreased yields, and inferior product quality. It is said to be more eco-friendly than other alkaline metal hydroxides such as sodium hydroxide (NaOH), but it's more expensive, hence less common. In applications involving environmental sustainability and recovery, activated carbon is an indispensable tool as it has larger responsibility as the globe continues to place a stronger concern on sustainability.

On the other hand, the ultimate and proximate analysis carried out by [4,9,13] indicates that shells and fibres of coconut have relatively high contents of fixed carbon and volatile matter with low ash content, and are ultimately environmentally friendly as a result of their low contents of sulphur and nitrogen. This work was aimed at optimizing the process parameters (activation temperature and impregnation ratio) of activated carbon production to determine the optimum responses (activated carbon yield and iodine adsorption capacities) using the response surface methodology (RSM) of the Design Expert statistical package.

RSM is a statistical tool used for the optimization of experimental processes. It uses quantitative data from suitably developed experimental designs to determine as well as solve multivariate problems, thus generating equations, which describe the effects of process parameters (independent variables) on responses (dependent variables). It also helps in the determination of interrelationships among response variables, representing their combined effect in any response. This method enables a researcher to conduct an effective study of an experimental process [14-16]. The optimization of industrial processes requires some response parameters that indicate the performance of systems to be optimized. To ensure the systems perform optimally, some variables are maximized, whereas others are minimized. In some cases, the dependent variables compete in such a manner that an increase in one response variable may adversely affect another, thus making the case to be more complicated [14,17]. Therefore, solving such a problem involves the use of approaches such as the constrained optimization procedure; the superimposition of

the contour diagrams of the different response variables; as well as using a desirability function that combines all dependent variables into one measurement [18].

2.0 MATERIALS AND METHODS

2.1 Sample Collection and Preparation /Instruments for Experimentation

The precursor for the preparation of bio-adsorbents (coconut coir) was a waste product obtained from the Enyiogugu market located in Imo State, southeastern Nigeria. The gathered coir was pretreated by washing it thoroughly with distilled water for the removal of impurities, and the samples were dried adequately in an oven at 40°C for a period of 14 hours [19]. The coir samples were then blended and sieved, and the particles which ranged from 2 to 3mm were taken and preserved in a desiccator for experimental purposes. Some quantities of the coir samples whose weights had been previously measured and recorded were placed a in 60% solution of potassium hydroxide (KOH) for 24 hours [19]. Then the carbonization of the coir samples was again conducted in a muffle furnace from 50°C to 500°C for 1 hour, washed using distilled water of pH 7, and dehydrated at 110°C for a period of 8 hours using the method suggested by [20]. The samples were subsequently sieved to the desired particle size using standard sieves of 2000µm, 2500µm and 3000µm, respectively. Other materials/instruments used for sample preparation and experimentation were a muffle furnace, pH/temperature meter, pyrometer, Bunsen burner, measuring cylinders (1000ml, 500ml, 100ml, and 10ml), milling machine and a stopwatch.

2.2 Methods

2.2.1 Sample Characterization

The physical and chemical characterization of coconut coir carbons was performed using the methods described in [21].

Tapped bulk density

This was obtained using the method proposed by [22]. A fixed mass (g) of the bio-adsorbent sample that was dehydrated at 110°C was placed in a 10 ml measuring cylinder. The base of the measuring cylinder was gently tapped on the top of the laboratory bench until no more change was observed in the level of the sample. Thereafter, the bulk density B_d was determined using Eq. (1).

$$B_d = \frac{W}{V} \quad (1)$$

where w is the weight of the dry sample, while v is the dry sample's volume.

Percentage Ash Content

The sample's percentage ash content A_c of the sample was determined by adding 3g of the pre-dried bio-adsorbent sample into crucibles that had been previously weighed and thereafter combustion in a muffle furnace at 550°C for a period of 5 hours in the presence of air [22]. The average value of three replicates was recorded and the value of A_c was obtained using Equation (2).

$$A_c = \left(\frac{W_{ash}}{W_{sample}}\right) \times 100 \quad (2)$$

where W_{ash} is the weight of ash, while W_{sample} is the weight of the bio-adsorbent sample.

Percentage Moisture Content

The determination of this property was achieved using the method proposed by [22]. Exactly 3g of the coir (carbonized) sample was weighed into the pre-weighed crucible and the total weight was taken. The sample was dried at 105°C for 5 hours, removed from the oven, cooled and repeatedly heated and weighed after 1 hour until a constant weight was obtained. The % moisture content was determined using Eq. (3).

$$\text{Moisture content} = \left(\frac{W_{sample} - W_{dry}}{W_{sample}}\right) \times 100 \quad (3)$$

where W_{sample} represents the weight of the bio-adsorbent sample prior to dehydration, and W_{dry} is the weight of the sample recorded after dehydration.

Pore Volume

This was determined using the methods suggested by Menkiti et al., [22]. Three grams of the bio-adsorbent was used. The sample was completely immersed in water and boiled until the air in the bio-adsorbent had been displaced. The sample was then superficially dried and

weighed. The pore volume was calculated using Equation (4).

$$V_{pore} = \left(\frac{W_{inc}}{\rho_{water}}\right) \times 100 \qquad (4)$$

where, V_{pore} is the pore volume of the bio-adsorbent and W_{inc} is the weight increase of the bio-adsorbent and ρ_{water} is the density of water.

Surface Area

The surface area was determined according to the modified methods of [20]. It was calculated from nitrogen gas adsorption isotherms based on Brunauer, Emmet and Teller's (BET) method of surface area analysis (at 77.305K) using a Quanta-chrome 2.0 analyzer.

2.2.2 Determination of Activated Carbon Yield and Adsorptive Capacities

Activated carbon Yield

In the preparation of activated carbon, its yield is defined mainly as the ratio of the final weight of activated carbon generated after activation, washing and dehydration, to the raw materials' initial weight; and both are on a dry basis [23].The activated carbon yield, R (%) was determined using Eq. (5):

$$R = \frac{m}{m_0} \times 100 \qquad (5)$$

where, m is the final weight of the dry activated carbon (g) and m_0 is the final weight of the dry precursor (g).

Adsorption Capacity of Iodine

Iodine was used as a probe molecule to assess the capacity of adsorbent in the adsorption of solutes of molecular sizes < 10Å. The definition of iodine number is considered to be the amount of iodine in milligrams that are absorbed by 1g of carbon. The adsorptive capacity of iodine was achieved using the volumetric method of sodium thiosulfate ($Na_2S_2O_3.5H_2O$) proposed by [23]. The estimation of the iodine number was actualized by the mixture of the activated carbon and 0.02N solution of iodine occasionally shaken, followed by titrating the solution against sodium thiosulfate using starch as an indicator.

Adsorption Capacity of Methylene Blue

Another probe molecule used for investigating the adsorbent's adsorptive capacity of solutes was methylene blue, but for molecular sizes >15Å. The methylene blue number is defined as the amount in milligrams of methylene blue adsorbed by 1g of carbon [23]. The methylene blue concentration was measured using a beam of Anthelie ultraviolet/visible light spectrophotometer data at the maximum wavelength of absorbance (660 nm).The adsorption capacities of methylene blue and iodine, Cads (mg/g) were obtained using Eq. (6):

$$Cads = \frac{(C_i - C_e)V}{m} \qquad (4)$$

where $C_i$and C_e (mg/L) represent the liquid-phase concentration of iodine/methylene blue at the initial and equilibrium conditions, respectively; whereas V represents the volume of the solution (L), and m represents the mass of the dry adsorbent employed (g).

2.2.3 Optimization of the carbon activation properties using response surface technique

This study considered desirability functions as indices for determining optimum experimental results. The adequacy of the generated model was checked using R^2, adjusted R^2 (Adj- R^2), Predicted R^2 (Pre-R2), Adeq Precision, and the coefficient of variance (C.V) values [16]. According to [21], the appropriateness of models is such that R^2> 0.95; Pre- R^2> 0.7; (Adj- R^2–Pre- R^2) < 0.2; Adeq Precision > 4**;** and C.V < 10. The Design Expert Software (version 6.0) was used in the Statistical Analysis of the results for the carbon activation properties. The experimental data were gathered in the required format for the statistical study, and the collated data were used in the analysis to generate the necessary statistical parameters useful in the statistical model development and optimization. Also, the software was employed in the response and contour plots of the experimental data which were also useful for the analysis and evaluation of the data variability.

Process Variables

The process variables (X) used for the experimental and statistical analysis was as follows:

X_1 = Activation temperature (^oC); X_2 = Impregnation ratio (g/g).

Response Variables

The response variables (Y_i) for the experimental and statistical analysis were:
Y_1 =Activated carbon yield (%)
Y_2 = Iodine adsorption capacities (mg/g).

Optimum Process Conditions

For the response variables; the optimum process conditions are the X_1 and X_2 values that yield the optimum (minimum) Y_i value.

Design of Experiment

Design Expert (DE) software was employed in the examination of the pattern of response as well as for the determination of the optimum activation temperature and impregnation ratio combination for optimizing the various carbon activation properties, for the various chemically treated carbons. The levels and range of optimized variables are presented in Table 1, while the experimental design code for the response variables is presented in Table 2. The central composite design helps to combine the hypercube vertices whose coordinates are represented using a factorial design of 2^n with star points. These star points help in estimating the nonlinear response surface curvature. The design of the experiment was actualized using Design Expert® 6.0, which resulted in 13 runs as shown in Table 3. These 13 experimental runs underwent randomization to maximize the observed responses, and the effects of unexplained variability.

Table 1: Actual and coded levels of factors for the 2-factorial central composite design

The symbols of independent variables		**Coded levels**				
		-2	-1	0	1	2
		Actual levels				
Activation temp (°C)	A	350	400	450	500	550
Impregnation ratio (g/g)	B	1	1.5	2	2.5	3

Table 2: Experimental design code for the responses (dependent variables)

1	Activated carbon yield (%)	Y_1
2	Iodine adsorption capacities (mg/g)	Y_2

3.0 RESULTS AND DISCUSSION

The results of the complete design matrix and the outcomes of the response variables generated from the experiments are presented in Table 3. Design Expert software was employed in the development of a correlation between the variables of activated carbon preparation and carbon response (yield).

Table 3: Experimental Design Matrix (with actual values; central composite design for the two factors and five levels)

S/N	**Run**	**Factor 1, X_1: Activation temp (°C)**	**Factor 2, X_2: Impregnation ratio (g/g)**	**Response, Y_1: Activated carbon yield (%)**	**Response, Y_2: Iodine adsorption capacities (mg/g)**
1	10	500	1.5	17.45	86.840
2	6	450	3	8.75	166.903
3	12	350	2	9.01	128.398
4	1	450	1	12.99	184.548
5	7	500	2	6.55	190.987
6	3	450	2.5	10.81	187.315
7	9	450	2	10.58	190.775
8	13	400	2	8.33	192.070
9	8	400	2.5	9.96	176.650

10	4	450	1.5	10.81	157.556
11	2	450	2	10.81	161.723
12	5	450	2	10.81	187.562
13	11	550	2	3.99	44.140

Table 4: Analysis of variance (ANOVA) for response surface quadratic model for Iodine adsorption capacity

ANOVA (Analysis of variance) table					
Source	**Sum of Squares**	**Df**	**Mean Square**	**F- Value**	**p-value**
Model	355.21	5	71.04	2220.81	<0.0001 Significant
A - Activation temperature	1.04	1	1.04	32.41	0.0007
B - Impregnation ratio	10.88	1	10.88	340.04	<0.0001
AB	4.95	1	4.95	154.72	<0.0001
A^2	252.82	1	252.82	7903.44	<0.0001
B^2	164.60	1	164.60	5145.56	<0.0001
Residual	0.2239	7	0.0320		
Lack of fit	0.0000	3	0.0746		
Pure Error	0.0000	4	0.0000		
Cor Total	355.43	12			

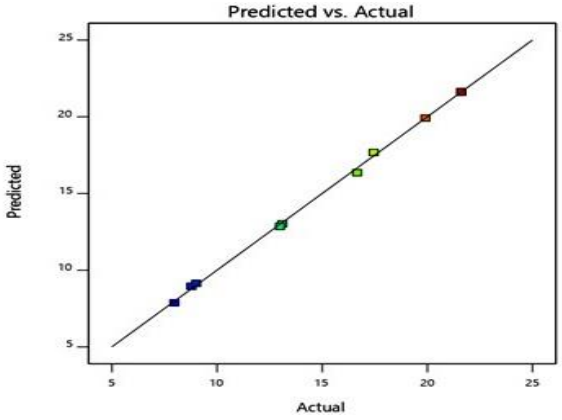

Figure 1: Predicted versus experimental for iodine adsorption capacity and carbon yield

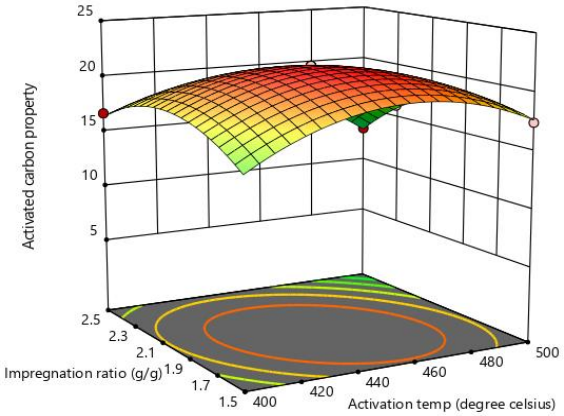

Figure 2: Three-dimensional response surface activated carbon yield plot of iodine (Effect of activation temperature and chemical impregnation ratio at t = 5 min).

Table 5: Optimization study (number of possible runs)

Number	Activation	Impregnation	R1	Desirability
1	354.592	1.420	4.830	1.000
2	384.684	2.991	7.481	1.000
3	481.250	3.000	5.785	1.000
4	495.469	2.987	3.817	1.000
5	373.215	2.989	6.062	1.000
6	350.117	1.399	3.232	1.000
7	386.250	1.000	4.483	1.000
8	399.961	1.016	7.527	1.000
9	350.332	1.604	6.185	1.000
10	394.136	1.030	6.762	1.000

3.1 DISCUSSION

Based on the results obtained from the adsorptive capacity of coconut fibre carbon, the developed model's quality was evaluated based on the coefficient of determination (R2), as well as the values of standard deviation. Small values of standard deviation as well as the negligible difference between the value of R2 and unity (1), show that the model can accurately predict the responses [16] and [25]. This further indicates that all the experimental variations can be absolutely explained by the response surface plot of the activated carbon property against impregnation ratio and activation temperature given in Figure 2. Similarly, the R2 value of 1 or close to unity illustrates perfect correlation with the values of the response variable parameters (activated carbon yield and iodine adsorption capacities) with the input variables (activation temperature and impregnation ratio). The adequacy of the models was further justified through analysis of variance (ANOVA). The ANOVA for the quadratic model for iodine adsorption capacity is presented in Table 4. From the ANOVA for the response surface quadratic model for iodine adsorption, the model F-value of 2220.81 implied that the model was significant. Values of p-values less than 0.01 indicated that the model terms were significant. From the statistical results obtained, it was shown that the developed model was adequate to predict the iodine capacity and the carbon yield within the range of variables studied.

Figure 1 shows the predicted values versus the experimental values for iodine adsorption capacity and carbon yield, respectively. As expected, the errors between the predicted and actual carbon yield were smaller as compared to the errors between the predicted and actual iodine adsorption capacity. The values of the carbon yield predicted were very close to the experimental values, thus showing that the developed model successfully captured the correlation between the carbon yield and the activated carbon preparation variables. Figure 2 shows the three-dimensional response surfaces which were plotted to show the interaction and effect of the activated carbon preparation variables (activation temperature and chemical impregnation ratio) on iodine (Y1). For this plot, the activation time was fixed at zero level (t = 5 min). As can be seen from Figure 2, the activated carbon property generally increases with an increase in activation temperature and chemical impregnation ratio.

In furtherance of the investigation of the adsorptive capacity of activated carbon described, the readings of best fit for all 10 possible runs (Table 5) were obtained not to be less than or greater than unity (1) with an activation temperature of 354.592oC, the quadratic model was selected, as expected. The closeness of the predicted values to corresponding experimental data is an indication of the accuracy of the quadratic model which explains the amount of carbon yield at given combination of the treatments.

For the final empirical models in terms of coded factors after excluding the carbon yield, the quality of the model developed was evaluated based on the correlation coefficient, R2 and also the standard deviation values. The closer the R2 value to unity and the smaller the standard deviation, the more accurate the response could be predicted by the model [26].

4.0 CONCLUSION

Activated carbon prepared with coconut fibre has been shown to contain a high degree of porosity and surface area. Due to various applications of activated carbons in water purification, wastewater treatment, and

pollutant removal, activated carbon prepared with coconut fibre can be used more for wastewater treatment due to its unique characteristics as evidenced in the results generated. The activations of activated carbon are done both physically and chemically with its yield ranging between 3.99% and 17.45 %. Compared to the physical activation method, chemical activation is more economical, because it requires a lower activation temperature, shorter activation time and a higher carbon yield [21,23]. The study explains the effectiveness of activated carbon prepared from coconut fibre in the adsorption of iodine. The iodine adsorption capacity varied between 44.140 and 192.070 mg/g. The use of potassium hydroxide as an activating agent gave a better result in terms of surface area and is more efficient in adsorption. It was observed that only the impregnation ratio and the treatment combination were statistically significant ($P < 0.0001$) to the determination of the carbon yield and iodine adsorption capacities. Further studies may consider the confirmation about the adsorption of some other unknown worthy pollutants that need to be removed from wastewater based on the calculated porosity of the coconut fibre.

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